

## **EPA Method 549.1**

### **Diquat and Paraquat from Drinking Water**

This method is a summary of the SPE extraction contained in section 11.2 “Cartridge Extraction” of US EPA method 549.1.

#### **1. Principle**

EPA 549.1 relies upon unusual ion pairing conditions that convert the reverse phase C8 into a solid support for the ion-pairing reagent, 1-hexanesulfonic acid (solution B). Also, during conditioning, cetyltrimethylammonium bromide (solution A) is used to deactivate residual silanol on the surface of the silica particle.

Note: Paraquat and Diquat absorb onto untreated glass surfaces. For this reason it is recommended to use plastic labware or to silanize all glass products that come into contact with the sample.

#### **2. Materials**

**Strata SPE Cartridge:** C8 1g/6mL , Part Number: 8B-S005-JCH

**Solution A:** Dissolve 0.5 g of cetyl trimethyl ammonium bromide and 5 ml of concentrated ammonium hydroxide in 500 ml of deionized water and Q.S. to 1 liter in a volumetric flask.

**Solution B:** Dissolve 10 g of 1-hexanesulfonic acid, sodium salt and 10 ml of concentrated ammonium hydroxide in 250 ml of deionized water and Q.S. to 500 ml in a volumetric flask.

**Elution Solution:** Add 13.5 ml of orthophosphoric acid and 10.3 ml of diethylamine to 500 ml of deionized water and Q.S. 1 liter.

#### **3. Specimen Preparation**

Adjust sample pH to 10.5 +/- 0.2 with 10% w/v NaOH or 10% v/v HCl. (Follow section 11.1 of EPA 549.1 for more detail if necessary. )

#### 4. Method

- **Condition:** *Sequentially pass through the cartridge:*

1. 1 column volume of Methanol
2. 1 column volume of DI Water
3. 1 column volume of Solution A. Allow to slowly percolate through the sorbent bed under low vacuum. ( 1 column volume in 1 - 2 minutes )
4. 2 column volumes of DI Water
5. 1 column volume of Solution B. Allow to slowly percolate through the sorbent bed under low vacuum. (1 column volume in 1 - 2 minutes). Allow 1-2 mm of Solution B to remain on top of the sorbent.

- **Load:**

Pass the sample through the cartridge. Maximum sample flow rate 6 ml per minute .

Note: Large volume samples may be processed by using an adapter cap (P/N AHO-7191) and 60 ml solvent reservoir (P/N AHO-7189) or attaching a solvent vacuum line to the sample collection bottle. Please refer to the Phenomenex document entitled “ Processing Large Volume Samples by SPE”

- **Wash:**

1. 1 column volume of DI Water
2. 1 column volume of Methanol.
3. Dry the cartridge under full vacuum for 30-60 seconds to insure removal of aqueous wash.

- **Elute:**

1. Place a collection container inside the manifold.
2. Pass 2 x 2.5 ml of Elution Solution through the cartridge at a rate not to exceed 3mL/min.

#### 5. Analysis

- A. Fortify eluent with 100 ul of ion-pair “ concentrate”. Concnetrate is made by dissolving 3.75 g of 1- hexanesulfonic acid in 25 ml of the Elution Solution.
- B. HPLC column is Hamilton PRP-1 or equivalent. Analyze by HPLC / DAD per Table 1, EPA 549.1.

Note: This information is designed to serve as a convenient summary of the solid phase extraction protocol contained in the referenced US EPA method. Phenomenex makes no guarantee regarding the accuracy or completeness of the method. Please contact the US EPA for a copy of the original, complete method.